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Fabrication of Cellulose Powder Dielectric Composite Material using Pineapple Leaves Fiber

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ABSTRACT

Pineapple, known for its delicious taste and nutritional value, generates substantial waste in the form of cores, leaves, and skin, resulting in significant yearly accumulations. Efficient disposal of pineapple waste has become a critical challenge due to its increasing production and potential environmental pollution. The objective of this paper is to volarize natural fiber derived from pineapple waste by fabricating it into a new dielectric composite material. The fabrication process of dielectric composite material was achieved by using the optimization technique through Design Expert software, resulting in noteworthy findings. Then, the properties of the fabricated materials were analyzed in terms of their permittivity value and elemental composition. The permittivity value of the newly fabricated dielectric materials was measured using the vector network analyzer (VNA) method, while their elemental composition was characterized using energy-dispersive X-ray (EDX) spectroscopy. The correlation between elemental composition and the permittivity value of newly fabricated composite materials is analyzed in this paper. As a result, the dielectric composite material attained its highest permittivity (4.08) when composed of 76.02% carbon and 22.61% oxygen. Conversely, the material exhibited a lower permittivity value (2.87) when its carbon content decreased to 69.32% and its oxygen content increased to 29.81%. This outcome highlights the crucial role of carbon in absorbing and storing electromagnetic signals, influencing the dielectric properties of the material. In conclusion, this paper unveils a ground-breaking use for waste pineapple leaves, showcasing how their carbon content significantly affects the resulting dielectric composite material's permittivity. This innovative eco-friendly material presents a sustainable alternative for non-recyclable dielectric materials in electronic devices such as PCBs, antennas, and sensors, for example.

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1. Introduction

Natural fibers from various sources find extensive use in materials engineering due to increased public awareness of conserving natural resources and developing eco-friendly products [1]. Natural fibers, being biodegradable and noncarcinogenic, are viewed as practical alternatives to synthetic fibers, and they offer better long-term availability [2]. In contrast, synthetic fibers, which are costlier and nonbiodegradable, contribute significantly to electronic waste generation [3]. A wide variety of plant-based sources, including banana leaves, coconut husks, palm kernel shells, oil palm fronds, sugarcane bagasse, rice husks, carrot waste, residues from the production of apple juice, coffee residues, and pineapple leaves, provide abundant natural fibers [4-10]. Among these, pineapple leaves, often discarded as waste after harvesting the fruit, boast promising chemical composition and mechanical properties that can be harnessed to produce valuable natural fibers [7]. Transforming pineapple leaves into dielectric material for electrical appliances is a sustainable approach that adds value to pineapple waste [7,11-15].

Natural fibers, mainly cellulose, hemicellulose, and lignin, with cellulose being the most common, are used as reinforcing agents [16]. Pineapple waste, particularly the cellulose-rich sections in its leaves, significantly enhances the non-essential strength and stiffness of composite materials, making it an excellent reinforcing agent [17]. Utilizing cellulose derived from pineapple waste as a filler in composite materials opens up possibilities for applications in electronics devices such as capacitors, insulators, printed circuit boards (PCBs), sensors, and diverse types of antennas [17-20]. This approach not only allows for the full utilization of natural resources but also contributes to the development of novel dielectric composite materials from pineapple waste [11]. A composite material is defined by the incorporation of diverse elements to achieve specific characteristics and functionalities. It may involve combining organic components, such as cellulose from pineapple waste, with other biologically sourced elements, leading to enriched material properties suitable for a wide array of applications in modern technology and engineering [7].

Characterizing the dielectric properties of newly developed composite materials holds vital significance for microwave communications, as it determines their signal absorption capacity [22]. An efficient electromagnetic absorber converts electromagnetic (EM) signals into heat [21-23]. The permittivity value (ε_r) of newly developed composite materials, represented through Eq. (1), reflects their capacity to absorb EM signals ($\varepsilon_{r'}$) and indicates the thermal energy value ($j\varepsilon_{r''}$) [25]. This essential parameter helps evaluate the ability of materials to absorb and store electromagnetic signals, while morphological analysis reveals the elemental compositions such as carbon (C), oxygen (O), sodium (Na), magnesium (Mg), calcium (Ca), and any other possible elements present in the fabricated composite material, which may become important factors influencing the material's performance [22].

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r" \tag{1}$$

Various factors are crucial to be considered when fabricating new dielectric composite material, including the use of different models of analysis such as one-factor at a time (OFAT), two-level factorial (TLFA), and optimization techniques [27,28]. For instance, optimization techniques are particularly valuable for maximizing the factors that affect the processing of new development material and minimizing the insignificant factors that can contribute to the performance of the fabrication material. In this paper, optimization techniques were employed to fabricate a new dielectric composite material using cellulose powder derived from pineapple leaves. Since the role and composition of pineapple waste vary significantly within the plant, individual analysis is necessary for optimizing the fabrication of new dielectric composite material [28-30].

Appropriate analysis techniques can provide a comprehensive understanding of the interactions among the contributing factors during fabrication. Based on the fabricated composite material, the correlation between the permittivity value and the elemental composition is studied. This knowledge holds the key to unlocking the optimum performance of the newly developed dielectric composite material. In addition, to the best of our knowledge, there has been limited exploration of permittivity and morphological analysis based on elemental composition in natural dielectric composite materials. Therefore, this study aims to fabricate a safe, eco-friendly, and green extraction technique for fabricating dielectric composite material using pineapple leaves and unveil the main elements that influence the fabricated material's performance.

2. Material and Methods

Figure 1 shows the overall phases involved in the fabrication of cellulose powder dielectric composite material using pineapple leaf fibers. The experiment was carried out in three phases. The first phase of the experiment was designed using the analysis model, which was optimization technique analysis. The optimization technique was used to obtain the desired design factors and the best set of operating conditions in order to execute the experimental work [31]. The second phase was dielectric material fabrication. The material fabrication employed pineapple leaves as the agricultural waste raw material. The third phase was material characterization. The characterization of the newly developed dielectric composite materials was analyzed in terms of their permittivity and elemental composition.

Fig. 1. Overall phases of cellulose powder dielectric composite material fabrication

2.1 Phase 1: Analysis Model

2.1.1 Optimization technique using design expert software

The fabrication of cellulose powder dielectric composite material using pineapple waste involves several factors that were analyzed and optimized using Design Expert software. Figure 2 shows the operation mechanism of the proposed analysis model. The factors, also known as analysis variables, are essential inputs for calculations in the experiment. Once all factor values are defined, the optimization technique evaluates the analysis model, generating outputs referred to as analysis functions or experiment responses. These functions serve as indicators of the design's quality [29]. By employing optimization techniques, a comprehensive understanding of the interactions among factors during the fabrication process of dielectric composite material using pineapple leaves can be achieved. This approach proves effective in optimizing resource utilization and minimizing environmental impact, ultimately guiding the identification of the most favorable experimental conditions [30].

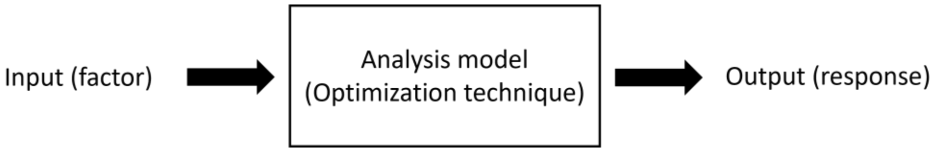


Fig. 2. Operation mechanism of the proposed analysis model [31]

The experiment was conducted according to the setup by following the standard order (STD), where all the factors were randomized. The TLFA technique was initially used to analyze important factors that could influence the fabrication of the dielectric material in our previous work [32]. In TLFA, the pulping time and the weight of the powdered cellulose from pineapple leaves were selected as the contributing factors that affect the increment in permittivity value [32]. The two selected factors, which are pulping time and the weight of the powdered cellulose from pineapple leaves, were chosen as factors in this experiment because these factors are showing significant changes. As the pulping time increases, the mixtures get more concentrated, which leads to an increase in the permittivity value of the material [32]. Meanwhile, another factor, which is the weight of the powdered cellulose from pineapple leaves, also shows a significant effect on the increment in permittivity value. When the weight of the powder increases, the amount of carbon in the fabricated dielectric composite material also increases, which at the same time increases the permittivity value of the fabricated dielectric composite material [32].

Thus, the pulping time and the weight of the powdered cellulose from pineapple leaves were then optimized by using optimization analysis techniques that run in Design Expert software. Optimization entails the modification of an existing process with the aim of enhancing the frequency of favorable outcomes while concurrently diminishing the incidence of undesirable outcomes. [18]. From this analysis, good-performance dielectric composite materials that can exhibit high permittivity values can be fabricated. Table 1 shows the optimization conditions that were designed and run by the Design Expert based on the 13 runs under STD. The experiment comprised 13 experimental runs, comprising 8 samples and 5 centre points. These experiments incorporated five levels of variation in numerical factors, specifically the axial points (plus and minus alpha), the factorial points (plus and minus 1), and the centre point. Alpha in Design Expert software refers to the minimum and maximum values of a factor, where plus alpha means the maximum value for the factor and alpha minus is the minimum value of the factor [18]. For the pulping time factor, the plus alpha used was 50 minutes and the minus alpha was 20 minutes, while for the factor weight of pineapple leaf powder, the plus alpha was 9 grams and the minus alpha was 1 gram. Even though the ratio of pineapple leaves to distilled water (PL:DW) and the heating option were analyzed in the TLFA result in [32], these two factors did not show a significant impact on the increment of permittivity values. The value of each run with its respective responses is shown in Table 2.

Table 1
Optimization conditions using Design Expert software

STD	Pulping time (minute)	Weight of pineapple leave powder (gram)
1	25	3
2	35	3
3	25	7
4	35	7
5	20	5
6	40	5
7	30	1
8	30	9
9	30	5
10	30	5
11	30	5
12	30	5
13	30	5

2.2 Phase 2: Material Fabrication

The pineapple leaves used in this study were collected from a pineapple plantation located in Pekan, Pahang, Malaysia, as shown in Figure 4(a). The leaves were harvested, purified to remove contaminants, and then dried for a week in the sunlight. A 2-cm-long portion was cut out of each of the dried leaves.

2.2.1 Cellulose extraction from pineapple leaves using chemical treatment

As shown in Figure 4(b), the cellulose of the pineapple leaves was extracted using a chemical treatment process, where sodium hydroxide (NaOH) was used in this experiment. This extraction method is referred to as the Kurschner-Hanack method, where this method will identify cellulose, hemicellulose, and lignin [33]. The leaves were treated with a 5 wt.% NaOH solution at a constant temperature of 100 °C. The solution was mixed with distilled water using a mass of pineapple leaves and a mass-to-solution ratio as calculated by Eq. (2) [32]. Chemical treatment improves interfacial adhesion between natural fibers and improves their mechanical, physical, and thermal properties [19]. Chemical treatment is also an effective complement to these compounds because it swells the cell wall of the matrix structure and improves its penetrability [19]. This treatment also removes hemicellulose, splits fibers into fibrils, and results in a closer-packed cellulose chain as a result of the relief of internal tension, thereby enhancing the mechanical properties of the fibers [19]. For untreated fiber, hemicellulose, lignin, pectin, waxy substances, and natural oils are present [16]. The chemically treated cellulose from pineapple leaves was used as filler, and epoxy resin was used as a polymer matrix to composite the dielectric material in this experiment. Based on the literature review, a polymer matrix is used in composite materials to hold the filler in place [33-35].

$$NaOH\ concentration\ (\%) = \frac{Mass\ of\ NaOH\ (g)}{Mass\ of\ distilled\ water\ (ml)} \times 100 \tag{2}$$

One hundred grams of pineapple leaves were initially boiled in a mixture of NaOH and distilled water, and different pulping times were used for the extraction process. The mixture was filtered using a mesh cloth filter and thoroughly washed before being squeezed until a clear effluent was obtained. The squeezed cellulose of pineapple leaves was dried under the sun for 1 week, as shown in Figure 4(c). The dried pulp was then ground to obtain cellulose-fiber pineapple leaf in the form of powder, as shown in Figure 4(d). Then the powder was mixed with Epox-Amite-100 Epoxy (laminating resin) and 102-Medium Epoxy (curative hardener) to fabricate a dielectric substrate, as shown in Figure 4(e and f). The benefit of fabricating dielectric composite materials through resin-based methods lies in their potential to extend their durability and lifespan when combined with epoxy. This effect arises from the inherent properties of epoxy, which can withstand substantial pressure and becomes nearly indestructible after curing due to its exceptional mechanical strength. Additionally, incorporating natural fibers in powdered form during the fabrication process has the potential to enhance material durability when integrated into the epoxy system [32]. Each sample has a different amount of powder, weighing 1, 3, 5, 7, and 9 grams.

2.3 Phase 3: Material Characterization

2.3.1 Permittivity measurements using VNA method

Figure 4(g) illustrates the setup for permittivity measurement of the fabricated dielectric material, involving a ROHDE & SCHWARZ ZNB-40-GHZ-Vector Network Analyzer (VNA) and a rectangular waveguide [37]. A preliminary calibration was executed on the VNA to mitigate potential measurement errors. The permittivity assessment was conducted within the G-band, spanning a frequency range from 3.95 to 5.85 GHz. To facilitate sample machining, the height of the sample matched that of the G-band waveguide at 22.15 mm. However, the width of the sample could vary within the waveguide limit of 47.55 mm. Ideally, the dielectric material was situated at the centre of the waveguide, harnessing field strength for precise detection of scattering wave changes. Connecting the waveguide to the VNA enabled measurement of the magnitude and phase of transmission coefficients. Importantly, this method of measurement omitted the utilization of the reflection coefficient due to its susceptibility to ambient noise during measurement, emphasizing the utilization of highly sensitive transmission coefficients.

Subsequently, the permittivity of the dielectric material was derived through the inverse method of the measured transmission coefficient [38]. Figure 3 shows the flow of the permittivity measurement. This involves calculating the transmission coefficient utilizing an electromagnetic theory fabrication, with an initial permittivity value estimated through iterative guessing. The iterative estimation procedure for the permittivity was reiterated until a satisfactory alignment between the measured and calculated transmission coefficient values was achieved. The adjustment of the permittivity value was carried out to minimize the disparity between the measured and calculated transmission coefficient parameters within the waveguide method. Once this disparity fell below the tolerance threshold of 0.1, the final guessed value of the permittivity was designated as the permittivity value of the dielectric composite material. This derived permittivity value from the last iteration serves as the foundational parameter for determining the material's dielectric constant, thereby characterizing its dielectric properties.

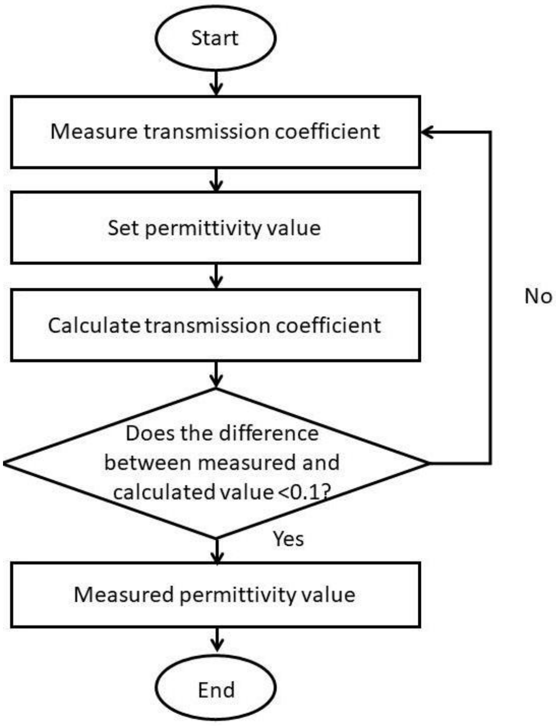
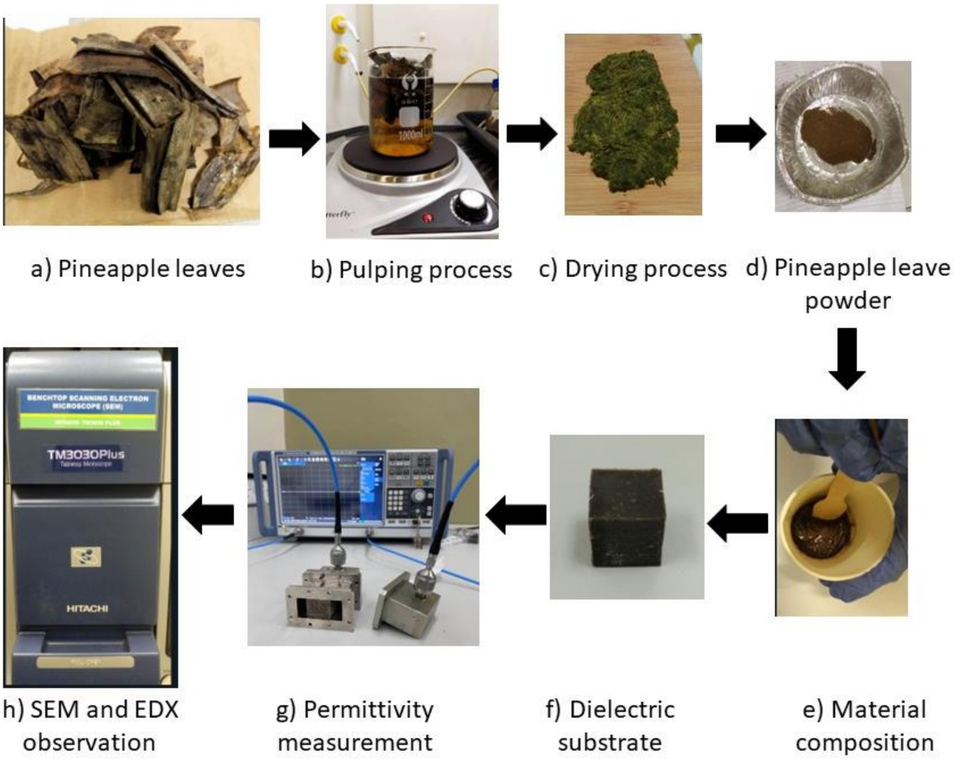


Fig. 3. Flow of the permittivity measurement

2.3.2 Analysis through morphological structure and elemental composition results

Energy-dispersive X-ray spectroscopy (EDX SEM; Hitachi/TM3030 PLUS, Japan) was used to analyze the elemental composition of the fabricated cellulose powder dielectric composite material from natural fiber pineapple leaves. The morphological structure was also observed as a supplemented result by using scanning electron microscopy (SEM; Hitachi/TM3030 PLUS, Japan) from the top view, as shown in Figure 4(h). This analysis was carried out in order to investigate the correlation between the permittivity value and the elemental composition of the material.



a) Pineapple leaves
b) Pulping process
c) Drying process
d) Pineapple leave powder
e) Material composition
f) Dielectric substrate
g) Permittivity measurement
h) SEM and EDX observation

Fig. 4. Experimental procedures include cellulose powder dielectric composite material substrate fabrication using pineapple leaves (a–f), permittivity value measurement (g), elemental composition analysis, and SEM observation (h)

3. Result and Discussion

3.1 Permittivity Value of Cellulose Powder Dielectric Composite Material

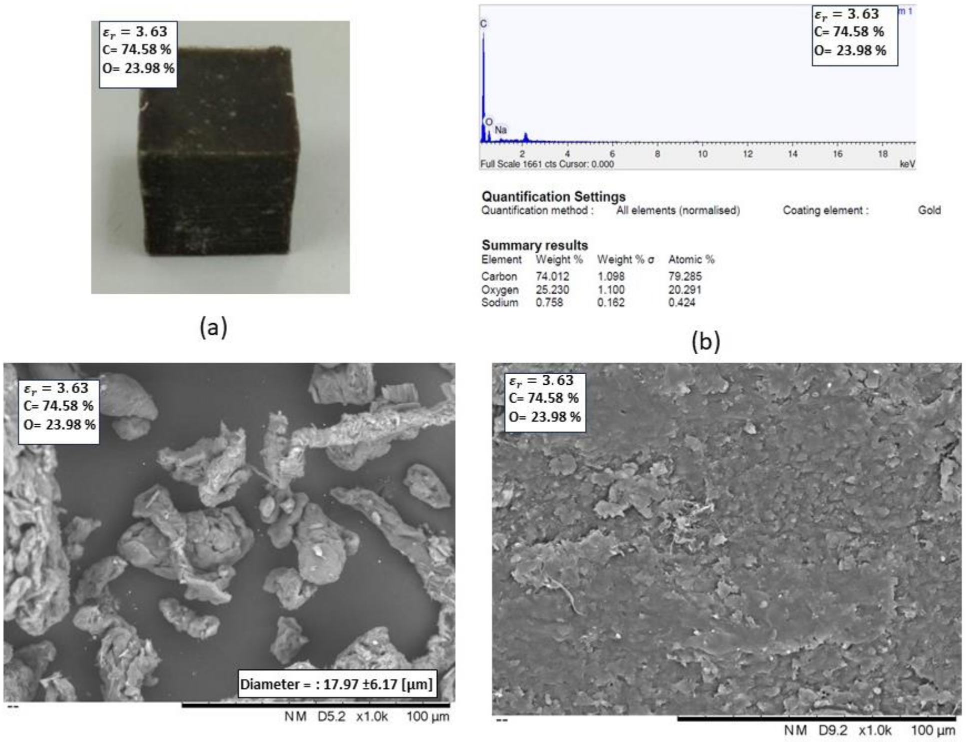
Table 2 presents the permittivity values, carbon percentage (%), and oxygen percentage (%) of dielectric composite materials that were fabricated from pineapple leaf cellulose. The experimental work was designed according to the STD order generated by Design Expert software using the optimization technique mentioned in the methodology section. Thirteen samples were fabricated under various factors, as shown in Table 1. The highest permittivity achieved was 4.08, while the lowest permittivity recorded was 2.87.

Table 2
Result of the permittivity value of the fabricated cellulose powder dielectric composite materials

STD	Permittivity value	Carbon percentage (%)	Oxygen percentage (%)
1	2.87	69.32	29.81
2	2.93	70.12	29.51
3	3.15	70.42	29.99
4	3.2	70.89	28.99
5	3.38	71.18	27.00
6	3.38	73.11	26.34
7	3.44	71.56	25.44
8	3.53	71.34	24.92
9	3.57	72.43	25.34
10	3.63	74.58	23.98
11	3.63	74.25	23.42
12	3.64	74.36	23.32
13	4.08	76.02	22.61

3.2 Morphology Image and EDX Analysis of The Cellulose Powder Dielectric Composite Material

Figure 5(a) shows the fabricated dielectric composite material with a size of 21.6 mm × 21.6 mm × 21.6 mm. The original color of the pineapple leaf powder exhibits a brownish hue; however, upon amalgamation with resin and epoxy, the resultant mixture undergoes a transformation, assuming a darker brownish shade. This phenomenon is likely attributable to a chemical reaction occurring between the pineapple leaf powder and the epoxy compound. The fabricated substrate was used for permittivity measurement, morphology observations, and elemental composition observations. Meanwhile, Figure 5(b) shows the EDX analysis for STD 10 as an example result of material, and Figure 5(c) shows the SEM image of the grinded pineapple leaf cellulose powder for the same STD 10. It reveals that the cellulose fibers derived from pineapple leaves exhibit irregular shapes, with an average particle size measuring 17.97 µm. Meanwhile, 6.17 µm indicated the standard deviation of the average particle size. The higher value of this standard deviation indicates that the data points are spread out over a large range of values [39]. Figure 5(d) shows the SEM image of the dielectric composite substrate for the same STD. The SEM images in Figures 5(c) and 5(d) were acquired at a magnification scale of ×1.0k, signifying a magnification factor of 1,000 times. The surface analysis of the dielectric composite substrate shown in Figure 5(d) indicates a smooth texture with seemingly no discernible particulate matter visible on the surface. It is hypothesized that these particles may have settled at the bottom of the epoxy layer, rendering them difficult to observe under SEM. Additionally, there is the potential for powder dilution during the fabrication process. However, while the presence of particles may not be readily discernible on the surface of the composite material, the question of whether the powder is effectively diluted during the fabrication process remains uncertain. This uncertainty underscores the significance of future research into the dynamics of particle dispersion in this material, making it a compelling avenue for investigation.



Quantification Settings

Quantification method : All elements (normalised) Coating element : Gold

Summary results

Element	Weight %	Weight % σ	Atomic %
Carbon	74.012	1.098	79.285
Oxygen	25.230	1.100	20.291
Sodium	0.758	0.162	0.424

(a) (b)

(c) (d)

Fig. 5. Fabricated cellulose powder dielectric composite of STD 10 (a), EDX analysis of dielectric composite material (b), SEM image of the grinded pineapple leaf (c), and SEM image of fabricated dielectric composite material from cellulose powder of the pineapple leaf (d)

3.3 Effect of Elemental Composition Changes with Permittivity Value

The EDX analysis of the pineapple leaf fiber shows that the pineapple leaf consists of carbon (C), oxygen (O), sodium (Na), silicon (Si), and chlorine (Cl) elements. Carbon and oxygen elements are the most significant elements in influencing the permittivity value of the dielectric composite material [32].

3.3.1 Analysis of carbon composition effects with permittivity value results

Figure 6 shows a comparison of the carbon element percentage and permittivity value. The graph shows that the lowest carbon percentage obtained was 69.32%, with a permittivity value of 2.87. In the meantime, the greatest permittivity value was 4.08, resulting in a carbon percentage of 76.02 %. The graph indicates that as the fraction of carbon elements grows, so does the permittivity value. F. Wee *et al.*, have stated that carbon plays an important role in absorbing or storing electromagnetic signals [26]. In other words, the permittivity value of the formulated dielectric material is influenced by its carbon content [40]. The permittivity of dielectric composites is correlated with their elemental content, with higher permittivity exhibited due to the presence of carbon [19]. This is due to the fact that carbon is a key component in helping the dielectric composite material absorb and store the electromagnetic signal [32]. The dielectric material absorbs and stores more electromagnetic impulses when there are more carbon elements present in the material. Conversely, the permittivity value of a dielectric material will be low if its carbon content is low. This is due to the fact that a dielectric composite material with a low carbon percentage will reduce the material's ability to store or absorb electromagnetic signals.

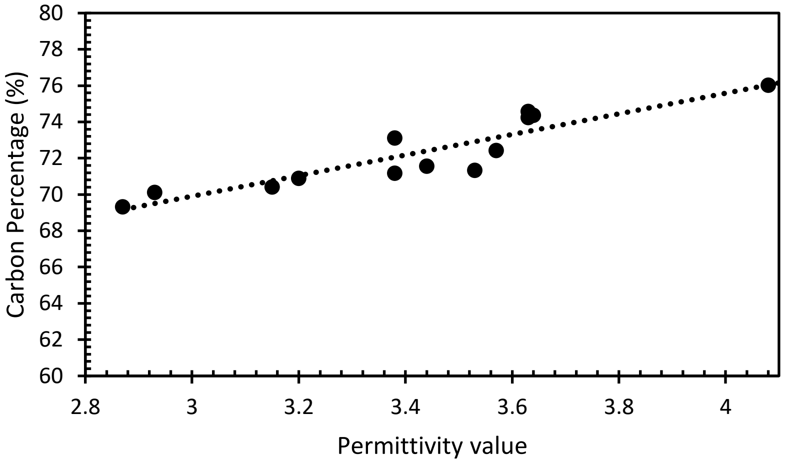


Fig. 6. Comparison between carbon percentage and permittivity value

3.3.2 Analysis of oxygen composition effects with permittivity value results

Figure 7 shows a comparison of the oxygen element percentage and permittivity value of the dielectric material. According to the graph, the maximum oxygen percentage obtained was 29.81%, with a permittivity value of 4.08. Meanwhile, with an oxygen percentage of 22.61%, the lowest permittivity value was 2.87. The graph indicates that when the amount of oxygen increases, the permittivity value decreases. According to S.S. Hossai *et al.*, as the oxygen element in the fabricated dielectric material increases, the permittivity value decreases [24]. This is because when the dielectric material has more oxygen, it will create more bubbles inside the dielectric material. The bubbles, also known as porous, are in circular vacuum areas filled with oxygen. Significantly, when there is more oxygen in the fabricated dielectric composite material, it will be more porous, resulting in the presence of void space and increasing the inability of the material to absorb or store electromagnetic signals. As a result, the permittivity value is reduced. When the oxygen percentage or porosity in the formed dielectric composite material is reduced, the permittivity value increases. In other words, as the amount of oxygen in the dielectric composite material increases, the permittivity value of the dielectric composite material will decrease [24].

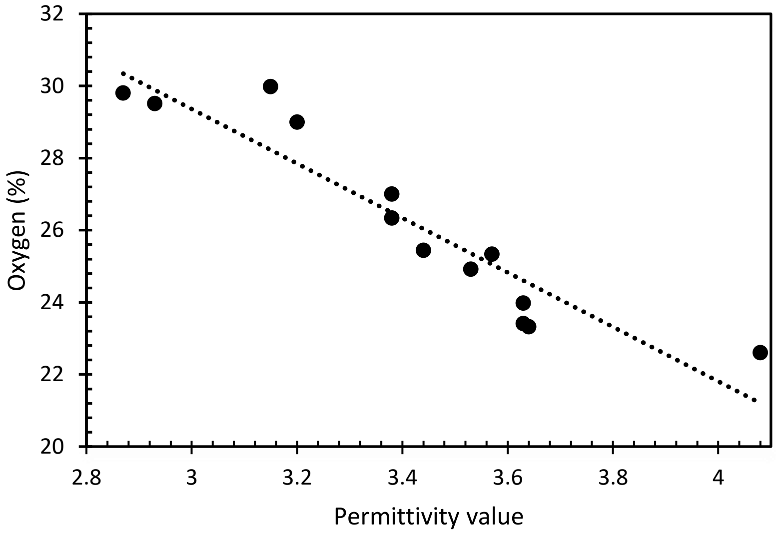


Fig. 7. Comparison between oxygen percentage and permittivity value

4. Conclusion

In this paper, the correlation between elemental compositions and the permittivity values of the newly fabricated dielectric composite materials from pineapple leaf fiber was well analyzed and discussed. The findings conclude that elemental composition considerably impacts the permittivity value of dielectric composite materials. The elements that significantly affect the permittivity value of the dielectric composite material are carbon and oxygen. Other elements, such as chlorine, sodium, and silicone, are not having much impact, and the percentage of each element in the composite material is significantly small and not consistent. On average, 70% of dielectric composite materials consist of carbon elements, and 30% are oxygen elements. A higher carbon content in the dielectric composite material enhances its capacity to absorb and store electromagnetic signals, resulting in an increase in the permittivity value of the fabricated material. This theory proved that when the permittivity value was 2.87, the carbon percentage was 69.32, and when the permittivity value was 4.08, the carbon percentage was 76.02. Besides that, oxygen also impacts the dielectric composite material's permittivity value. This is because when the oxygen element forms pores in the material, it reduces its ability to absorb and store the electromagnetic signal. This occurred when the oxygen percentage was 29.81 and the permittivity value was 2.87, and when it was 22.61, the permittivity value was 4.08. Therefore, in this study, we proved that pineapple fiber can be used as a valuable product by fabricating a dielectric composite material. We understood that the permittivity value of fabricated dielectric composite material depends on its elemental content and fabrication factors, which may influence material performance. Producing enormous amounts of dielectric composite materials from the natural fiber of pineapple leaves mitigates electronic waste pollution due to their ability to biodegrade. For future recommendations, adding different ingredients from different types of natural fiber from different types of plants to the formulation of a new composite may improve the dielectric properties of the desired fabricated dielectric composite material, allowing it to be employed in a wide range of electrical applications.

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